

## AN INITIAL STUDY ON ANTIMONY LEACHING FROM LEAD CONCENTRATE

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**ABSTRACT.** The work confirms that (i) lead-zinc (Pb-Zn) polymetallic ores in the Rhodopes region contain antimony (Sb) in concentrations higher than the Clarke number and (ii) the flotation beneficiation of these ores concentrates Sb in Pb concentrate. The initial leaching experiments with 2 M nitric acid, 2 M sulphuric acid alone and mixed with sodium chloride (1 M), and 2 M sodium sulphide (Na<sub>2</sub>S) in 1 M sodium hydroxide (NaOH) solutions were carried out at a temperature of 90-95 °C. Only 2 M Na<sub>2</sub>S in 1M NaOH solution practically does not leach lead. Initial treatment of the material with a solution of sulphuric acid containing sodium chloride, followed by neutralisation and contacting with an alkaline sodium sulphide solution is pointed (at a proof-of-concept level) as an possible approach for antimony leaching, with minimal leaching of lead.

**Key words:** antimony, leaching, lead concentrate.

### ПЪРВОНАЧАЛНО ПРОУЧВАНЕ НА ИЗЛУЖВАНЕТО НА АНТИМОН ОТ ОЛОВЕН КОНЦЕНТРАТ

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**РЕЗЮМЕ.** Работата потвърждава, че (i) оловно-цинковите (Pb-Zn) полиметални руди в района на Родопите съдържат антимон (Sb) в концентрации, по-високи от числото на Кларк, и (ii) флотационното обогатяване на тези руди концентрира Sb в Pb концентрат. Проведени са първоначални експерименти за излужване с 2 M азотна киселина, 2 M сярна киселина самостоятелно и смесена с натриев хлорид (1 M) и 2 M натриев сулфид (Na<sub>2</sub>S) в 1 M разтвор на натриев хидроксид (NaOH) при температура 90-95 °C. Само 2 M Na<sub>2</sub>S в 1M разтвор на NaOH практически не излужва олово. Първоначалното обработване на материала с разтвор на сярна киселина, съдържащ натриев хлорид, последвано от неутрализация и третиране с алкален разтвор на натриев сулфид, е посочено (на ниво доказване на концепцията) като евентуален възможен подход за излужване на антимон, с минимално излужване на олово.

**Ключови думи:** антимон, излужване, оловен концентрат.

### Introduction and theoretical background

Antimony (Sb) is used as a flame retardant in rubber, plastic, textiles, paper, etc. Along with its compounds, it is applied as a stabiliser and catalyst in the chemical industry. One of the main uses of Sb is in production of high mechanical strength lead-antimony (Pb-Sb) alloys applied in automotive lead-acid batteries and in construction (Nishad and Bhaskarapillai, 2021; Dembele et al., 2022; U.S. Geological Survey, 2026).

Recently, antimony and its compounds have been studied as materials suitable for application in renewable energy sources, such as solar cells, lithium and sodium ion batteries (Parize, et al., 2017; Nie et al., 2021; Moolayadukkam et al., 2022; Wu et al., 2024).

Antimony supply is at high risk due to the geographical concentration (sometimes in politically unstable countries) of the natural ore deposits and global production. The leading country of antimony production is China, ensuring 36.4% of the world's total production for 2025. The reserves are concentrated in China (41%), Russia (17%), and Bolivia (15%) (U.S. Geological Survey, 2026). The Sb supply vulnerability increases because it is recovered as a by-product from lead, copper, silver, and gold minerals. An increasing demand for antimony and its compounds is expected and if the present extraction rate is maintained, it is anticipated that the extractable global resources of Sb will be exhausted by 2050 (Henckens et al., 2016).

All of the above explain why antimony is included in the 2025 USA list of critical raw materials (CRMs) (<https://www.usgs.gov/programs/mineral-resources-program/science/about-2025-list-critical-minerals>), as well as in the 2023 European Union list of CRMs (European Commission, 2023).

Stibnite (Sb<sub>2</sub>S<sub>3</sub>) is the most important antimony mineral from an industrial point of view. Other Sb-bearing minerals of

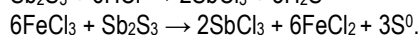
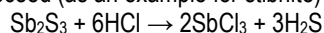
industrial interest are jamesonite (Pb<sub>4</sub>FeSb<sub>6</sub>S<sub>14</sub>), tetraedrite (Cu<sub>12</sub>Sb<sub>4</sub>S<sub>13</sub>), zinkenite (Pb<sub>9</sub>Sb<sub>22</sub>S<sub>42</sub>), boulangerite (Pb<sub>5</sub>Sb<sub>4</sub>S<sub>11</sub>), bindheimite (Pb<sub>2</sub>Sb<sub>2</sub>O<sub>6</sub>(O,OH)), as well as lead sulphide ores (Anderson, 2012; Dembele, 2022).

Antimony is extracted from its primary resources through pyrometallurgy or hydrometallurgy. The particular pyrometallurgical process used depends on the grade of the Sb concentrate to be treated (Anderson, 2019; Moosavi-Khoonsari, 2022).

Antimony is recovered from low-grade ores and industrial process residues by hydrometallurgical technologies (Anderson, 2019). Aqueous solutions and proper chemical reactions are used to leach valuable metals in the pregnant leach solution (PLS). After the PLS purification, the aimed metals are recovered through diverse chemical and physicochemical processes.

Hydrometallurgical recovery of Sb employs mainly two lixiviant types - acidic chloride containing systems and alkaline sulphide systems (Anderson, 2001).

Mainly HCl solutions are used by the industry, often together with ferric chloride (FeCl<sub>3</sub>). The following generalised reactions proceed (as an example for stibnite) (Ye, 2019):



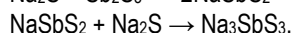
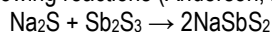
Ferric chloride is used as an oxidising and chloridising agent that transforms the antimony sulphide mineral into a chloride complex.

However, HCl solution and its mixture with FeCl<sub>3</sub> are highly corrosive (Noor and Al-Moubaraki, 2008; Nwokocha and Burleigh, 2024). In addition, these solutions are able to effectively dissolve galena (Dutrizac, 1986; Baba and Adekola, 2011; Nnanwube et al., 2018).

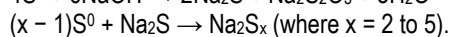
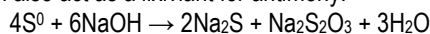
Different complexes are obtained at 25 °C - PbCl<sub>n</sub><sup>2-n</sup> (0 ≤ n ≤ 4) - while at elevated temperatures of 300 °C the predominant

species are  $\text{PbCl}^+$ ,  $\text{PbCl}_2^0$ , and  $\text{PbCl}_3^-$  (Seward, 1984; Luo and Millero, 2006).

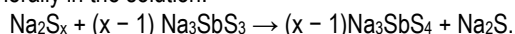
A mixture of sodium sulphide ( $\text{Na}_2\text{S}$ ) and sodium hydroxide ( $\text{NaOH}$ ) is the lixiviant used in the alkaline leaching. Sodium thioantimonite ( $\text{Na}_3\text{SbS}_3$ ) is formed as described by the following reactions (Anderson, 2001):



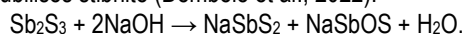
Elemental sulphur, naturally available in minerals, dissolves in  $\text{NaOH}$  to form sulphur species (sulphide -  $\text{S}^{2-}$ , sodium thiosulphate -  $\text{Na}_2\text{S}_2\text{O}_3$ , and sodium polysulphides -  $\text{Na}_2\text{S}_x$ ) that can also act as a lixiviant for antimony:



Polysulfide oxidises sodium thioantimonite to sodium thioantimonate ( $\text{Na}_3\text{SbS}_4$ ) that is the main species found generally in the solution:



The addition of  $\text{NaOH}$  prevents  $\text{Na}_2\text{S}$  hydrolysis and also solubilises stibnite (Dembele et al., 2022):



Although the alkaline sulphide leaching is considered very selective for antimony, it still possesses some drawbacks. According to some studies (Multani et al., 2016), when Sb minerals occur with galena, sphalerite, pyrite,  $\text{NaOH}$  is able to leach these minerals to form correspondingly the soluble plumbite ( $\text{Na}_2\text{PbO}_2$ ), zincate ( $\text{Na}_2\text{ZnO}_2$ ), and iron hydroxide ( $\text{Fe}(\text{OH})_2$ ) that can cause a certain loss of the reagent.

In industry and the predominating part of the studies devoted to Sb leaching from low-grade Sb minerals or from industrial waste, the Sb concentrations are considerably higher or co-measurable with the lead concentrations in the studied raw material. However, if the Sb is in materials containing high lead concentrations and very low Sb concentrations, as is the case with Pb concentrate, then the task is to leach the antimony into an aqueous solution, avoiding considerable leaching of lead from the concentrate.

In nature, Sb is often interconnected with ores of lead and zinc base metals (Reuter et al., 2019). Some Bulgarian geological studies point out the Sb presence in lead-zinc ores in the Rhodopes (Dimitrov, 1988; Marchev, et al., 2005; Popov and Popov, 2019).

Antimony often concentrates alongside lead in galena (lead sulphide) concentrate. This occurs because antimony frequently forms mixed sulphide or "sulphosalt" minerals (such as boulangerite or tetrahedrite) that naturally float alongside galena during the ore processing and concentration phases (Cao, et al., 2018; Megaloudi et al., 2021).

It is known that galena ( $\text{PbS}$ ) dissolves in nitric acid ( $\text{HNO}_3$ ) due to the acid's highly oxidising nature, converting lead sulphide into soluble lead nitrate, nitrogen oxides, and elemental sulphur (Halikia, et al., 2002; Strunnikov and Koz'min, 2005). However, the oxidation of sulphide sulphur to elemental sulphur and sulphate may cause certain passivation of the galena surface (Pashkov et al., 2002; Nnanwube and Onukwuli, 2025). The aqueous oxidation of galena studied in autoclave experiments with nitric acid at different concentrations (0.31–0.91 M) and temperatures (130–170 °C) showed the galena passivation by a small amount of anglesite ( $\text{PbSO}_4$ ) on its surface (Aydoğan, et al., 2007).

Galena does not dissolve directly in sulphuric acid, it undergoes a surface oxidation reaction that forms an insoluble,

passivating film of lead sulphate ( $\text{PbSO}_4$ ) that effectively halts further dissolution (Xie et al., 2021; Xie et al., 2022).

However, it is widely believed that  $\text{H}_2\text{SO}_4$  is not a suitable leaching reagent for low-grade Sb ores (Anderson, 2001).

By contrast, Rahimzoda and coauthors (Rahimzoda et al., 2024) state that treatment with 1.80 M  $\text{H}_2\text{SO}_4$  solution at a solid to liquid (S/L) ratio = 1 : 6 and a temperature of 80 °C, leaches 85% of the Sb from a solid pyrometallurgical product obtained by calcination of Sb-containing materials with  $\text{NaCl}$  for 2 h. When the  $\text{HCl}$  solution was used, 75% of the Sb available in the same solid material was leached under the same conditions. We consider that probably the introduction of chlorides in the system during the chlorination roasting process improved the results.

All of the above leads us to the idea to study (i) whether Sb from Bulgarian lead-zinc / polymetallic ores is concentrated in the galena concentrate, and (ii) what is the ability of  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{SO}_4$  with the addition of  $\text{NaCl}$ , and  $\text{Na}_2\text{S}$  –  $\text{NaOH}$  solutions to leach Sb from the galena concentrate, while dissolving as less lead as possible.

## Materials and methods

Ore, lead concentrate, zinc concentrate, and flotation tailings, obtained from a lead-zinc industry, are used in the study, with an emphasis on galena concentrate. Chemical analyses of the samples were performed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The solid samples were digested with the Aqua Regia - IMS40B method. The liquid samples were analysed using the standard addition method. In both cases, certified reference materials were used in calibrations. The analysis of the solid samples was performed by SGS BG Company and of the liquid samples - by the Educational and Scientific Laboratory of Trace Analysis: ISP Techniques and Radioanalytical Methods – “St. Kliment Ohridski University”. The lead concentrate, that was further used in leaching experiments, was subjected to an X-Ray Diffraction study at the corresponding Laboratory at the University of Mining and Geology “St. Ivan Rilski”. Bruker D2 Phaser was used at  $\text{Cu}$ ,  $\lambda = 1,54184$ , 30 kV, 10 mA, Two Theta 5°–90°, Increment 0,02° and 4191 steps.

In leaching experiments, a concentrate with particles of size +75 - 150  $\mu\text{m}$  was used. Solutions of  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{Na}_2\text{S}$ ,  $\text{NaOH}$ , and  $\text{NaCl}$  were prepared using p.a. grade chemicals and distilled water. The solid to liquid ratio was = 1 : 5. Experiments were carried out in 300 mL glass reactors, ensuring simultaneous temperature maintenance and stirring with a magnetic stirrer, as well as condensation of the vapours and return of water using a reflux condenser. The experimental conditions were chosen based on the literature (Anderson, 2001; Yang, 2017; Dembele et al., 2024) and presented in Table 1. For each condition, two parallel experiments were conducted and the results presented below are averaged.

Table 1. *Experimental conditions*

| Expt. type | Conditions                                                                                                                                                                  |
|------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1          | 2M $\text{HNO}_3$ , 2 h, 50-55 °C                                                                                                                                           |
| 2          | 2M $\text{H}_2\text{SO}_4$ , 2 h, 50-55 °C                                                                                                                                  |
| 3          | 1 M $\text{NaCl}$ in 2M $\text{H}_2\text{SO}_4$ , 2 h, 50-55 °C                                                                                                             |
| 4          | 2 M $\text{Na}_2\text{S}$ in 1M $\text{NaOH}$ , 2 h, 90-95 °C                                                                                                               |
| 5          | 1 M $\text{NaCl}$ in 2M $\text{H}_2\text{SO}_4$ , 2 h, 50-55 °C<br>washing until neutral reaction followed by 2 M $\text{Na}_2\text{S}$ in 1M $\text{NaOH}$ , 2 h, 90-95 °C |

The amount of extracted Sb (in %) is calculated using equation (1):

$$\text{Extracted Sb, \%} = (\text{Sb}_{\text{in liquid}} / \text{Sb}_{\text{in solid}}) \times 100 \quad (1)$$

## Results and discussion

Table 2 presents the concentrations of Pb, Zn, and Sb in the different material streams determined by ICP-MS analysis of the solid phase.

Table 2. Concentration of studied metals in the different material streams

| Material       | Sb, ppm | Pb, % | Zn, % |
|----------------|---------|-------|-------|
| Ore            | 9.97    | 2.89  | 2.03  |
| Pb concentrate | 149.2   | > 65  | 5.80  |
| Zn concentrate | 7.8     | 3.80  | > 60  |
| Tailings       | 4.41    | 0.22  | 0.52  |

The data shown in Table 2 confirm that the lead-zinc ores from the studied region contain Sb in amounts higher than its average estimated abundance in the Earth's crust, which is in the range from 0.2 to 0.5 ppm (<https://www.usgs.gov/centers/national-minerals-information-center/antimony-statistics-and-information>). It is clear that Sb is concentrated in lead concentrate – nearly 15-fold in this specific case. The results from the X-Ray analysis showed that the studied lead concentrate was composed mainly of galena, but other components were also present - ore phases were represented

by sphalerite, pyrite and chalcopyrite, and non-mineral phases - by kaolinite and quartz (Fig. 1). Most probably, due to the low amounts of Sb in the materials, its minerals have not been detected by the XRD analysis.

The results from the leaching experiments, described in Table 1, are presented in Table 3.

Table 3. Results from the leaching experiments

| Exp. type | Extracted metal, % |          |
|-----------|--------------------|----------|
|           | Sb                 | Pb       |
| 1         | 0.003              | 1.08     |
| 2         | 0.0023             | 0.0018   |
| 3         | 0.0022             | 0.05     |
| 4         | 5.8                | 0.000003 |
| 5         | 21.9               | 0.000002 |

As can be seen from the data given in Table 3, HNO<sub>3</sub> acid solution leaches a very low amount of Sb but a high amount of Pb, showing that the galena surface is not passivated. The finding may be assigned to the presence of pyrite in the sample. Zárate-Gutiérrez et al. (2012) proved that the pyrite presence modified both the reaction mechanism and the minerals dissolution kinetics, accelerating the oxidation of galena by depassivating its surface.

The percentages of leached antimony and lead with a 2 M solution of sulphuric acid are not only very low, but also approximately the same. This suggests that perhaps the destruction of the crystal lattice of galena releases isomorphously included antimony.

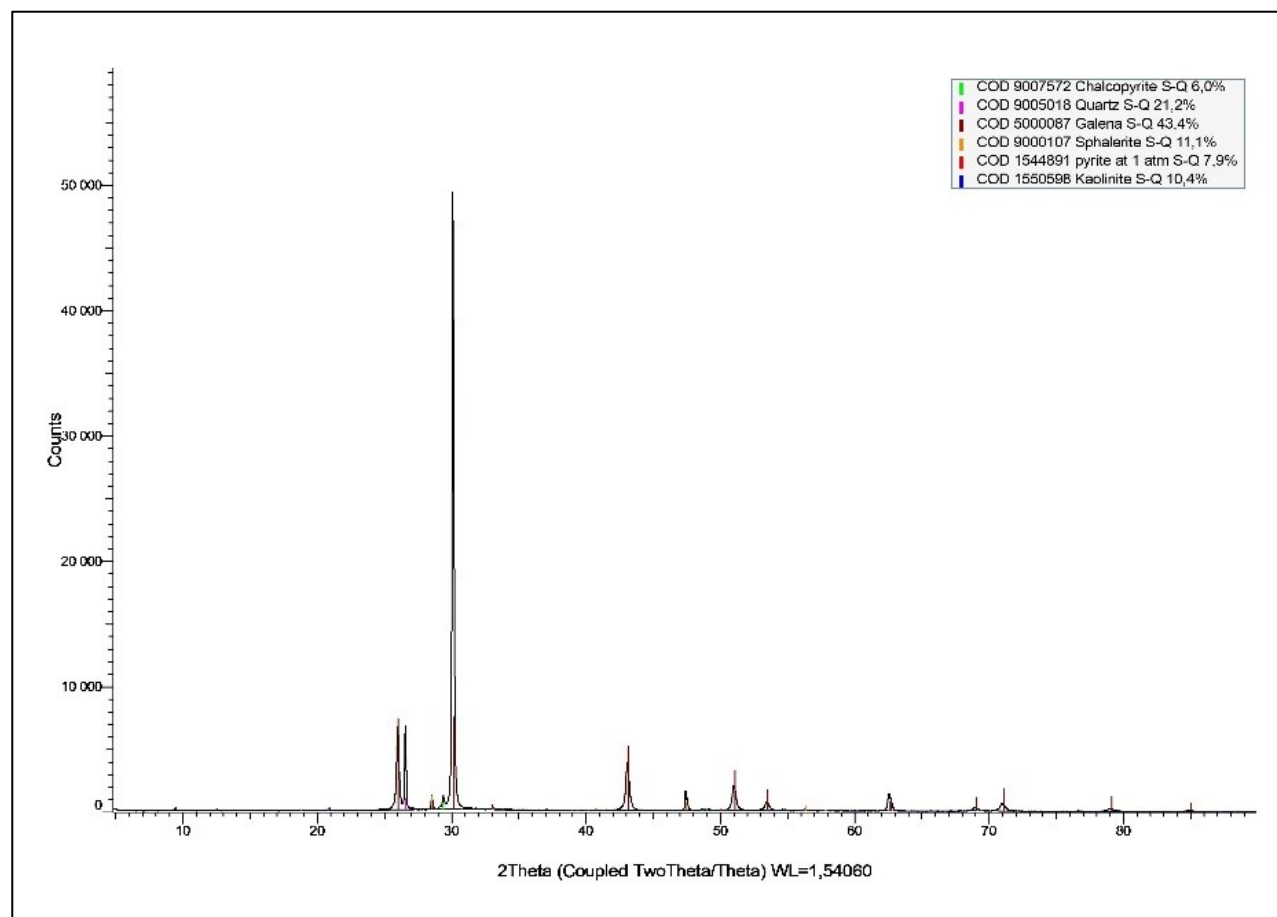


Fig. 1. Diffractogram of the used lead concentrate

The addition of sodium chloride to the sulphuric acid solution significantly increases the leaching of lead - about 28 times, while practically not changing the leaching of antimony.

Dembele and coauthors also found a relatively low leaching ability of even more concentrated 4 M solutions of  $\text{H}_2\text{SO}_4$  and  $\text{HNO}_3$  towards stibnite – 26 and 18 % correspondingly, at a temperature of 90 °C, when stibnite flotation tailings were leached and the impeding lead was practically not present in the raw material (Dembele et al., 2024). As the best leaching solution in their case, they found 4.4 M HCl that is not applicable when the raw material is mainly galena.

The use of a sulphide-alkaline solution under the conditions of the experiment practically does not leach lead, while the leaching of antimony is significant - experiment 4 – Table 3. Megaloudi et al. (2021) proposed leaching in a sodium sulphide strongly alkaline solution to extract Sb from galena concentrate containing Pb (52.89%), S (17.39%), Sb (9.91%), Fe (4.47%), As (2.79%), and Zn (1.73%). However, their concentrate included Sb in a relatively high concentration, under the form of boulangerite and jamesonite. They found that the increase in  $\text{Na}_2\text{S}$  concentration in the leaching solution and temperature caused an increase in Sb extraction, both factors increasing the dissolution rate. In addition, an increase in the S / L ratio led to a decrease in Sb extraction. About 97-98 % of the antimony available were dissolved within 2 h with a solution containing 1.25 M NaOH and 0.96 M  $\text{Na}_2\text{S}$  at 98 °C, but from Sb own ores that occurred in the Pb concentrate. The X-ray patterns showed that boulangerite peaks disappeared because the mineral was dissolved.

On the other hand, the initial treatment of the sample with a solution of sulphuric acid and sodium chloride, followed by washing and treatment with an alkaline solution, increased the extraction of antimony nearly 4 times, while practically did not further dissolve lead, compared to that leached during the initial treatment of the sample with the sulphuric acid solution. This could be an approach to intensify antimony leaching, while reducing lead leaching. All of the above hints that a certain pre-treatment of a galena concentrate with Cl-ion bearing sulphuric acid solution followed by alkaline leaching could enhance Sb leaching and deserves further future experiments. The finding is partially in line with discoveries of Rahimzoda and coauthors' (Rahimzoda et al., 2024), who achieved 85% leaching of Sb that was present in the solid pyrometallurgical product obtained by calcination with the NaCl of Sb-containing materials followed by leaching for 2 h with 180 g/L  $\text{H}_2\text{SO}_4$  solution at a S/L ratio = 1: 6 and a temperature of 80 °C.

## Conclusions

Based on the studies carried out, the following conclusions can be drawn:

1. It is confirmed that lead-zinc polymetallic ores in the Rhodopes region bare Sb in concentrations higher than the average Clarke number.
2. During the flotation beneficiation of lead-zinc ores, Sb is concentrated in Pb concentrate.
3. At this stage of the research, for the purpose of antimony leaching, with minimal leaching of lead, an initial treatment of the material with a solution of sulphuric acid containing sodium chloride, followed by the material leaching with a solution containing sodium sulphide and sodium hydroxide is outlined as a possible approach for treating the lead

concentrate. The results obtained should be considered only as proof of concept. For the implementation of the process, a number of further experiments with varying the leaching conditions (lixiviant concentrations and ratio of dissolved reagents, S/L ratio, temperature, stirring rate, particles size of the solid material) are needed in order to find the optimal ones.

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## References

- Anderson, C. G. (2001). Hydrometallurgically treating antimony-bearing industrial wastes. *JOM*, 53, 18–20. <https://doi.org/10.1007/s11837-001-0156-y>
- Anderson, C. G. (2012). The metallurgy of antimony. *Geochemistry*, 72, 3–8. DOI: 10.1016/j.chemer.2012.04.001
- Anderson, C. G. (2019). Antimony Production and Commodities. In R.C. Dunne, S. Komar-Kawatra, & C.A. Young (Eds.) *SME Mineral Processing and Extractive Metallurgy Handbook*. Society for Mining, Metallurgy and Exploration, Inc. (pp. 1557–1568). Englewood, CO, USA
- Aydoğan, S., Erdemoğlu, M., Uçar, G., & Aras, A. (2007). Kinetics of galena dissolution in nitric acid solutions with hydrogen peroxide. *Hydrometallurgy*, 88, 52–57. doi:10.1016/j.hydromet.2007.03.005
- Baba, A. A., & Adekola, F. A. (2011). Comparative analysis of the dissolution kinetics of galena in binary solutions of  $\text{HCl}/\text{FeCl}_3$  and  $\text{HCl}/\text{H}_2\text{O}_2$ . *International Journal of Minerals, Metallurgy, and Materials*, 18(1), 9–17. <https://doi.org/10.1007/s12613-011-0393-1>
- Cao, Q., Chen, X., Feng, Q., & Wen, S. (2018). Activation mechanism of lead ion in the flotation of stibnite. *Minerals Engineering*, 119, 173–182. <https://doi.org/10.1016/j.mineng.2018.01.039>
- Dembele, S., Akcil, A., & Panda, S. (2022). Technological trends, emerging applications and metallurgical strategies in antimony recovery from stibnite. *Minerals Engineering*, 175, Article 107304. <https://doi.org/10.1016/j.mineng.2021.107304>
- Dembele, S., Akcil, A., & Panda, S. (2024). Investigation of the characteristics of stibnite ( $\text{Sb}_2\text{S}_3$ ) flotation tailings and extraction of critical metals (Sb and As): Optimisation and scale-up. *Minerals Engineering*, 216, Article 108883. <https://doi.org/10.1016/j.mineng.2024.108883>
- Dimitrov, R. (Ed.) *Olovno-tsinkovite nahodishta v Balgaria*, Tehnika, Sofia, 1988.
- Dutrizac, J. E. (1986). The dissolution of galena in ferric chloride media. *Metallurgical Transactions B*, 17, 5–17.
- European Commission (2023). COM(2023) 160 Final ANNEXES to the Proposal for a Regulation of the European Parliament and of the Council Establishing a Framework for Ensuring a Secure and Sustainable Supply of Critical Raw Materials and Amending Regulations (EU) 168/2013, (EU) 2018/858, 2018/1724 and (EU) 2019/1020. European Commission. Brussels, Belgium.
- Halikia, I., Zoumpoulakis, L., & Christodoulou, E. (2002). Lead sulphide leaching kinetics in nitric acid solutions. *Erzmetall* 55(3), 166–175.
- Henckens, M. L. C. M., Driessen, P. P. J., & Worrell, E. (2016). How can we adapt to geological scarcity of antimony?

- Investigation of antimony's substitutability and of other measures to achieve a sustainable use. *Resources, Conservation and Recycling*, 108, 54–62. <https://doi.org/10.1016/j.resconrec.2016.01.012>
- Luo, Y., & Millero, F. J. (2006). Stability constants for the formation of lead chloride complexes as a function of temperature and ionic strength. *Geochimica Cosmochimica Acta*, 71(2), 326–334. doi: 10.1016/j.gca.2006.09.019
- Marchev, P., Kaiser-Rohrmeier, M., Heinrich, C., Ovtcharova, M., von Quadt, A., & Raicheva, R. (2005). Hydrothermal ore deposits related to post-orogenic extensional magmatism and core complex formation: The Rhodope Massif of Bulgaria and Greece. *Ore Geology Reviews*, 27, 53–89. <https://doi.org/10.1016/j.oregeorev.2005.07.027>
- Megaloudi, R. A., Kourtis, A., Oustadakis, P., Tzamos, E., Dimitriadis, D., & Xenidis, A. (2021). Antimony Extraction from Galena Concentrates. *Materials Proceeding*, 5, 130. <https://doi.org/10.3390/materials2021005130>
- Moolayadukkam, S., Bopaiah, K. A., Parakkandy, P. K., & Thomas, S. (2022). Antimony (Sb)-Based Anodes for Lithium-Ion Batteries: Recent Advances. *Condensed Matter*, 7(1), Article 27. DOI: 10.3390/condmat7010027
- Moosavi-Khoonsari, E., Mostaghel, S., Siegmund, A., & Cloutier, J. P. (2022). A Review on Pyrometallurgical Extraction of Antimony from Primary Resources: Current Practices and Evolving Processes. *Processes*, 10(8), Article 1590. <https://doi.org/10.3390/pr10081590>
- Multani, R. S., Feldmann, T., & Demopoulos, G. P. (2016). Antimony in the metallurgical industry: A review of its chemistry and environmental stabilisation options. *Hydrometallurgy*, 164, 141–153. 10.1016/j.hydromet.2016.06.014
- Nie, R., Hu, M., Risqi, A. M., Li, Z., & Seok, S. I. (2021). Efficient and stable antimony seleniodide solar cells. *Advanced Science* 8, Article 2003172.
- Nishad, P.A., & Bhaskarapillai, A. (2021). Antimony, a pollutant of emerging concern: A review on industrial sources and remediation technologies. *Chemosphere*, 277, Article 130252. <https://doi.org/10.1016/j.chemosphere.2021.130252>
- Nnanwube, I. A., & Onukwuli, O. D. (2025). Kinetics of galena leaching in nitric acid and sodium chloride solutions for potential lead recovery. *Canadian Metallurgical Quarterly*, 64(4), 2399–2414. <https://doi.org/10.1080/00084433.2025.2452779>
- Nnanwube, I. A. Onukwuli, O. D., & Sunday, U. A. (2018). Modelling and Optimisation of Galena Dissolution in Hydrochloric Acid: Comparison of Central Composite Design and Artificial Neural Network. *Journal of Minerals and Materials Characterisation and Engineering*, 6(3). DOI: 10.4236/jmmce.2018.63021;
- Noor, E. A., & Al-Moubaraki A. H. (2008). Corrosion Behavior of Mild Steel in Hydrochloric Acid Solutions. *International Journal of Electrochemical Science*, 3(7), 806–818. [https://doi.org/10.1016/S1452-3981\(23\)15485-X](https://doi.org/10.1016/S1452-3981(23)15485-X)
- Nwokocho, T., & Burleigh, T.D. (2024). Corrosion Resistance of 316L Stainless Steel in HCl and FeCl<sub>3</sub>. In: TMS 2024 153rd Annual Meeting & Exhibition Supplemental Proceedings. TMS 2024. The Minerals, Metals & Materials Series. Springer, Cham. pp 1205–1217 [https://doi.org/10.1007/978-3-031-50349-8\\_105](https://doi.org/10.1007/978-3-031-50349-8_105)
- Parize, R., Katerski, A., Gromyko, I., Rapenne, L., Roussel, H., Karber, E., Appert, E., Krunks, M., & Consonni, V. (2017). ZnO/TiO<sub>2</sub>/Sb<sub>2</sub>S<sub>3</sub> core-shell nanowire heterostructure for extremely thin absorber solar cells. *Journal of Physical Chemistry C*, 121(18), 9672–9680. <https://doi.org/10.1021/acs.jpcc.7b00178>
- Pashkov, G. L., Mikhlin, E. V., Kholmogorov, A. G., & Mikhlin, Y. L. (2002). Effect of potential and ferric ions on lead sulphide dissolution in nitric acid. *Hydrometallurgy*, 63, 171–179. 10.1016/S0304-386X(01)00221-3
- Popov, K., & Popov, P. (2019). The Alpine late collisional Rila-Rhodope Metallogenic Zone of the Balkan Orogenic System. *Review of the Bulgarian Geological Society*, 80, 1, 55–79.
- Rahimzoda, K. S., Anderson, C. G., Badalov, A. B., Kadirov, A. A., & Eshov, B. B. (2024). Hydrometallurgical Processing of Antimony Bearing Pyrometallurgical Calcines. *Journal of Mineral and Material Science (JMMS)*, 5(5), Article 1097. DOI: 10.54026/JMMS/1096
- Reuter, M. A., van Schaik, A., Gutzmer, J., Bartie, N., & Abadias-Llamas, A. (2019). Challenges of the Circular Economy: A Material, Metallurgical, and Product Design Perspective. *Annual Review of Materials Research*, 49, 253–74. <https://doi.org/10.1146/annurev-matsci-070218-010057>
- Seward, T. M. (1984). The formation of lead(II) chloride complexes to 300°C: A spectrophotometric study, *Geochimica et Cosmochimica Acta*, 48(1), 121–134. [https://doi.org/10.1016/0016-7037\(84\)90354-5](https://doi.org/10.1016/0016-7037(84)90354-5)
- Strunnikov, S. G., & Koz'min, Yu. A. (2005). Hydrometallurgical schemes of lead concentrate processing. *Chemistry for Sustainable Development*, 13(4), 483–490.
- U.S. Geological Survey (2026). *Mineral commodity summaries 2026 (ver. 1.3, May 2026)*. U.S. Geological Survey. <https://doi.org/10.3133/mcs2026>.
- Wu, Y., Shuang, W., Wang, Y., Chen, F., Tang, S., Wu, X. L., Bai, Z., Yang, L., & Zhang, J. (2024). Recent Progress in Sodium-Ion Batteries: Advanced Materials, Reaction Mechanisms and Energy Applications. *Electrochemical Energy Reviews*, 7, Article 17. DOI <https://doi.org/10.1007/s41918-024-00215-y>
- Xie, H., Jin, Y., Zhang, P., Liu, Y., Gao, L., Feng, Q., & Liu, D. (2022). Surface modification mechanism of galena with H<sub>2</sub>SO<sub>4</sub> and its effect on flotation separation performance. *Applied Surface Science*, 579, Article 152129. <https://doi.org/10.1016/j.apsusc.2021.152129>
- Xie, H., Liu, Y., Rao, B., Wu, J., Gao, L., Chen, L., & Tian, X. (2021). Selective passivation behavior of galena surface by sulphuric acid and a novel flotation separation method for copper-lead sulphide ore without collector and inhibitor. *Separation and Purification Technology*, 267, Article 118621. <https://doi.org/10.1016/j.seppur.2021.118621> ;
- Yang, T., Rao, S., Liu, W., Zhang, D., & Chen, L. (2017). A selective process for extracting antimony from refractory gold ore. *Hydrometallurgy*, 169, 571–575. <https://doi.org/10.1016/j.hydromet.2017.03.014>
- Ye, L., Ouyang, Z., Chen, Y., & Chen, Y. (2019). Ferric chloride leaching of antimony from stibnite. *Hydrometallurgy*, 186, 210–217. 10.1016/j.hydromet.2019.04.021
- Zárate-Gutiérrez, R., Lapidus, G. T., & Morales, R. D. (2012). Aqueous oxidation of galena and pyrite with nitric acid at moderate temperatures. *Hydrometallurgy*, 115–116, 57–63 DOI:10.1016/J.HYDROMET.2011.12.010. <https://www.usgs.gov/programs/mineral-resources-program/science/about-2025-list-critical-minerals> <https://www.usgs.gov/centers/national-minerals-information-center/antimony-statistics-and-information>